Kayrobertsonite, MnAl$_2$(PO$_4$)$_2$(OH)$_2$·6H$_2$O, a new phosphate mineral related to nordgauite

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Abstract: Kayrobertsonite, MnAl$_2$(PO$_4$)$_2$(OH)$_2$·6H$_2$O, is a new secondary phosphate mineral from the Hagendorf Süd pegmatite, Hagendorf, Oberpfalz, Bavaria, Germany and the Foote Lithium Company mine, Kings Mountain district, Cleveland County, North Carolina, USA. Kayrobertsonite crystals occur as intergrown masses of snow-white, soft, finely fibrous needles, less than 5 μm in diameter and no more than 100 μm in length. Quantitative analysis of Foote mine kayrobertsonite gave the empirical formula: Mn$_{0.97}$Ca$_{0.03}$Fe$_{0.02}$Al$_{1.87}$(PO$_4$)$_2$(OH)$_{1.62}$F$_{0.03}$(H$_2$O)$_{0.38}$·6H$_2$O; and for Hagendorf Süd kayrobertsonite: Mn$_{0.92}$Ca$_{0.06}$Fe$_{0.02}$Al$_{1.87}$(PO$_4$)$_2$(OH)$_{1.19}$F$_{0.42}$(H$_2$O)$_{0.39}$ · 6H$_2$O. Foote mine kayrobertsonite crystals are biaxial (–), with indices of refraction α = 1.530(1), β = 1.554(1), γ = 1.566(1), measured in white light; 2V(meas.) is 70.3(5)°, while 2V(calc.) is 69.6°. The mineral is nonpleochroic. The orientation of the crystals is Z ≈ **c** (length slow). Kayrobertsonite is triclinic, space group $P\bar{1}$, with the unit-cell parameters: a = 10.049(2), b = 10.205(2), c = 6.083(1) Å, α = 91.79(3), β = 99.70(3), γ = 98.02(3), V = 607.9(2) Å^3 and Z = 2 (Foote mine). The polyhedral framework in kayrobertsonite has the same topology as that in nordgauite, but with replacement of F by OH at the bridging anion sites. The main crystal chemical change from nordgauite to kayrobertsonite is a doubling of the number of water molecules in the [001] channels.

Key-words: kayrobertsonite; new mineral; nordgauite; pegmatite; Foote Lithium Company mine; Hagendorf; crystal structure; synchrotron; phosphate.

1. Introduction

The new mineral kayrobertsonite was first noted by Birch *et al.* (2011) as 'fibrous nordgauite' at the Hagendorf South pegmatite (Hagendorf-Süd), Hagendorf, Oberpfalz, Bavaria, Germany (49°39'1''N, 12°27'35''E). They noted that ''. . . *Calculation of the empirical formula on the same basis shows that OH is dominant over F in the site they share. The soft fibrous form is, therefore, the OH-analogue of nordgauite and represents a new species.*'' Due to lack of material, further investigations on 'fibrous nordgauite' were not possible. Subsequent to this discovery, one of the authors (JBS) noted that fibrous white needles found on the East dump of the Foote Lithium Company mine, Kings Mountain district, Cleveland County, North Carolina, USA (35°12'40''N, 81°21'20''W) matched this description and, based on this material, the description of the new mineral could be completed.

The two occurrences are co-type localities and here we report data for both occurrences.

The name honours German–American mineral collector Gabriella K. (Kay) Robertson (b. 1920) of Los Angeles, California, USA. Since the mid-1950s, Kay has been an ardent and sophisticated mineral collector, specialising in German minerals. The German portion of her 13 000+ specimen collection is among the most extensive and comprehensive in private hands. The phosphate minerals of Hagendof-Süd, her favourite German locality, are particularly well represented. Kay has always been generous in providing specimens and expertise to curators and mineral researchers. Her knowledge of old German mineral localities and her extensive reference library have made her a sought-after resource for deciphering old labels. Her contribution to scientific mineralogy of which she is most proud is her insightful detective work that was crucial in solving the ''xanthoxenite problem'' (Moore & Ito, 1978). Further information about Kay Robertson and her

collection can be found in her collector profile in *The Mineralogical Record* (Moore, 2007). Kay Robertson has agreed to the naming of the mineral in her honour.

The mineral and name (IMA2015–029) were approved by the IMA CNMNC prior to publication. Two cotype specimens from the Foote mine and one from Hagendorf are housed in the collections of Museum Victoria, registered numbers M53379, M53380 and M48795, respectively. Two cotype specimens from the Foote mine are housed in the collections of the Natural History Museum of Los Angeles County, registration numbers 65561 and 65562. Specimen M48795 is also one of the cotype specimens of nordgauite.

2. Occurrence, location and physical and optical properties

At Hagendorf, the new mineral was collected by EK in 1975–6, between the 60 and 67-m levels of the mine (Keck, 1983). The specimens consist of fragments of etched, pinkish brown zwieselite–triplite containing irregular cavities up to a few cm across. Here, kayrobertsonite occurs within the cavities along with fluorapatite, nordgauite, white-ite-CaMnMn, members of the jahnsite group, morinite, fluel-lite, Al-bearing strunzite and an analogue kingsmountite.

At the Foote mine, kayrobertsonite was found in a very large spodumene-bearing pegmatite boulder. The mineral occurred in small vugs in, or adjacent to, highly altered spodumene laths in the pegmatite body. There was no primary phosphate mineralization present and the occurrence of this paragenesis was highly localized, as is typical for the phosphate assemblages at the Foote Mine. Kayrobertsonite was found associated with (in order of decreasing abundance): mangangordonite, variscite, eosphorite, kastningite, paravauxite, beraunite, strengite, strunzite and cacoxenite. Vugs bearing the minerals are no larger than about 1 cm^3 and are usually much smaller. Kayrobertsonite, like nordgauite, has formed as a late-stage hydrothermal phase, likely to be one of the last phases formed in a complex suite of Al-rich secondary phosphates.

At both localities, kayrobertsonite crystals occur as intergrown masses of snow-white, soft, finely fibrous needles, less than 5 μm in diameter and no more than 100 μm in length (Figs. 1 and 2). The thin needles are elongated along [001] and flattened on {100}. Crystals are translucent with a silky lustre, and are somewhat flexible with a splintery fracture. No cleavage was observed. The Mohs' hardness is estimated to be about 2, based upon behaviour when broken.

The density (Foote mine), measured by the sink/float method in sodium polytungstate solution with water, is 2.29(3) g/cm^{-3}. The calculated density is 2.41 g cm^{-3}, based on the empirical formula and single-crystal unit cell. Optically, Foote mine kayrobertsonite crystals are biaxial (–), with indices of refraction α = 1.530(1), β = 1.554(1) and γ = 1.566(1), measured in white light. The 2V angle measured using extinction data analysed

Fig. 1. Fibrous bundles of kayrobertsonite with kastningite from the Foote mine. Field of view (FOV) approximately 8 mm across. JBS specimen #5979. (online version in colour)
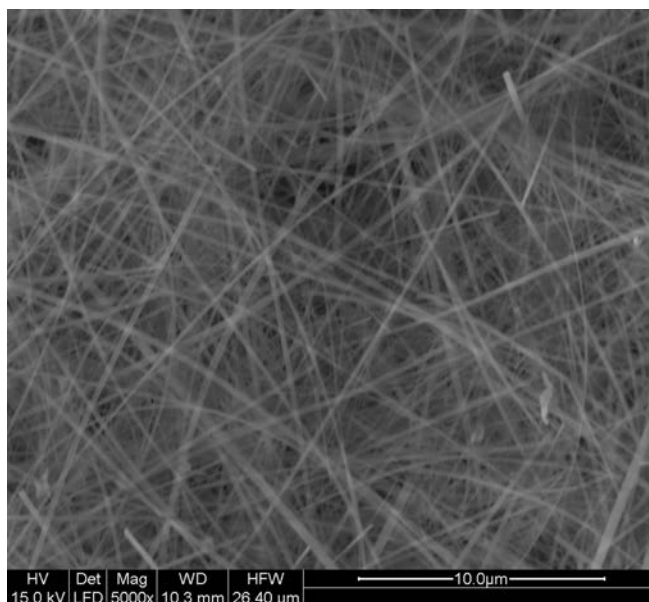

Fig. 2. Secondary electron image of kayrobertsonite needles from Hagendorf. FOV approximately 30 μm across.

with EXCALIBR (Gunter *et al.*, 2004) is 70.3(5)°, while 2V(calc.) is 69.6°. Dispersion could not be observed and the mineral is nonpleochroic. The orientation of the crystals is $Z \approx \mathbf{c}$ (length slow); X and Y orientations were not determined. The Gladstone–Dale compatibility is –0.030 (excellent) using the empirical formula and single-crystal unit cell.
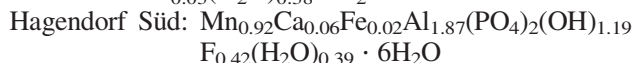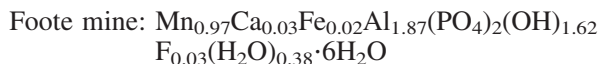
3. Chemical composition

Eight quantitative chemical analyses of kayrobertsonite from both localities were carried out by means of an

Table 1. Analytical data for kayrobertsonite.

Constituent	Foote mine wt.%	SD	Hagendorf Süd wt.%	SD	Probe standard
CaO	0.41	0.12	0.74	0.16	Wollastonite
MnO	16.11	0.58	15.20	0.59	Rhodonite
FeO	0.27	0.21	0.30	0.21	Hematite
Al_2O_3	22.43	0.88	22.17	0.49	$MgAl_2O_4$
P_2O_5	33.36	0.84	33.11	1.60	$AlPO_4$
F	0.13	0.17	1.88	0.30	CaF_2
$-O = F$	-0.05		-0.79		
H_2O_{calc}	29.30		29.30		
Total	101.96		101.91		

electron microprobe (WDS mode, on a JEOL JXA 8500F Hyperprobe, 12 kV, 2 nA and 2 µm defocused beam). Water could not be analysed directly because of the minute amount of sample available. H_2O was calculated on the basis of $6H_2O + 2(OH,H_2O)$ per formula unit (*pfu*). Analytical data are given in Table 1. The empirical formulae, with OH adjusted for charge balance are:

Foote mine: $Mn_{0.97}Ca_{0.03}Fe_{0.02}Al_{1.87}(PO_4)_2(OH)_{1.62}$
$F_{0.03}(H_2O)_{0.38}\cdot 6H_2O$
Hagendorf Süd: $Mn_{0.92}Ca_{0.06}Fe_{0.02}Al_{1.87}(PO_4)_2(OH)_{1.19}$
$F_{0.42}(H_2O)_{0.39} \cdot 6H_2O$

The simplified formula is $MnAl_2(PO_4)_2(OH)_2\cdot 6H_2O$. It is interesting to note that the Foote mine material is near-endmember, whereas the Hagendorf material contains significant but subordinate F. This indicates that a complete solid solution is likely to exist between kayrobertsonite and nordgauite.

4. Powder X-ray diffraction

X-ray powder diffraction data on the Foote mine kayrobertsonite were obtained using a Rigaku R-AXIS Rapid II curved imaging plate microdiffractometer, with monochromatized Mo$K\alpha$ radiation. A Gandolfi-like motion on the φ and ω axes was used to randomize the sample and observed d spacings and intensities were derived by profile fitting using JADE 2010 software. Triclinic cell parameters refined from the powder data using JADE 2010 with whole pattern fitting are: $a = 10.114(4)$, $b = 10.225(4)$, $c = 6.100(4)$ Å, $\alpha = 91.864(14)$, $\beta = 99.747(14)$, $\gamma = 98.471(11)°$, $V = 613.9(4)$ Å^3 and $Z = 2$.

Synchrotron X-ray powder diffraction data on the Hagendorf kayrobertsonite were obtained using the powder diffraction beamline at the Australian Synchrotron. A wavelength of 0.9543 Å was employed. The unit-cell parameters were obtained from whole pattern fitting using FULLPROF. Refined cell parameters are: $a = 10.060(3)$, $b = 10.167(2)$, $c = 6.108(1)$ Å, $\alpha = 91.79(3)$, $\beta = 99.99(3)$, $\gamma = 98.48(2)$, $V = 607.5(9)$ Å^3 and $Z = 2$. The powder data for both localities are given in Table 2.

Table 2. Powder X-ray diffraction data for kayrobertsonite. The powder pattern calculated from the structure determination (at 100 K) is included for comparison.

Hagendorf I_{obs}	d_{obs}	Foote I_{obs}	d_{obs}	Structure d_{calc}	I_{calc}	hkl
100	10.149	100	10.047	10.1911	100	0 1 0
		8	9.804	9.9060	7	1 0 0
36	7.700	44	7.629	7.6922	45	1̄ 1 0
		2	6.529	6.6317	2	1 1 0
		2	5.591	5.6119	1	1̄ 0 1
		3	5.296	5.3347	1	0 1̄ 1
21	5.011	12	5.029	5.0724	7	0 1 1
		4	4.958	4.9797	3	1̄ 1 1
		4	4.816	4.8544	4	1̄ 1̄ 1
15	4.736	10	4.695	4.7376	8	2̄ 1 0
		3	4.514	4.5538	3	1 1̄ 1
				4.2819	4	1 2 0
11	4.207	4	4.209	4.2172	11	2 1 0
		8	4.154	4.1599	5	1 1 1
		3	4.107			
5	4.058	5	3.991	4.0107	7	2̄ 1 1
3	3.849			3.8461	3	2̄ 2 0
				3.7171	2	1̄ 2 1
				3.5408	6	2 0 1
13	3.490	7	3.499	3.4988	3	2 1̄ 1
		3	3.463	3.3970	1	0 3 0
				3.3693	5	1̄ 3 0
13	3.303	5	3.324	3.3158	9	2 2 0
		8	3.259	3.2864	1	3̄ 1 0
				3.2093	2	2 1 1
				3.1163	2	2̄ 2 1
18	3.063	12	3.023	3.0772	18	1 3 0
				3.0368	3	0 3 1
23	2.992	10	2.952	2.9785	17	3̄ 2 0
				2.9438	3	0 1̄ 2
				2.9401	2	1 3̄ 1
		8	2.924	2.9250	1	1̄ 1̄ 2
25	2.901	6	2.891	2.9050	9	1̄ 1 2
				2.8680	2	1̄ 3̄ 1
		8	2.831	2.8530	7	0 1 2
20	2.811	5	2.793	2.8060	5	2̄ 0 2
				2.7163	2	3 1̄ 1
				2.7064	3	3 0 1
9	2.674	6	2.646	2.6674	6	0 2̄ 2
				2.6265	4	1̄ 2̄ 2
				2.6030	2	1 1 2
13	2.597	10	2.579	2.5977	3	1̄ 2 2
11	2.546			2.5393	3	2̄ 3 1
				2.4922	2	4̄ 1 0
				2.4003	2	2 1̄ 2
4	2.380			2.3682	1	1 4̄ 1
				2.2862	2	3 3̄ 1
4	2.263			2.2657	2	3̄ 2 2
				2.1961	5	0 3 2
5	2.155	5	2.139	2.1582	5	4̄ 3 0
				2.1077	3	4 2̄ 1
				2.0055	4	0 4̄ 2
				1.9910	4	2̄ 0 3
17	1.9870	6	1.982	1.9812	4	5 0 0
				1.9801	2	3 3 1
		4	1.958	1.9532	2	2̄ 1̄ 3
10	1.9438			1.9408	1	1 5 0
				1.9384	2	5̄ 2 1
				1.9104	2	1 0 3

Table 2. Continued

Hagendorf		Foote		Structure		
I_{obs}	d_{obs}	I_{obs}	d_{obs}	d_{calc}	I_{calc}	hkl
7	1.8890	2	1.896	1.8932	1	5 1 0
				1.8789	4	$\bar{3}\,\bar{4}\,1$
				1.8585	2	$\bar{2}\,\bar{4}\,2$
9	1.8476	3	1.849	1.8502	2	1 1 3
		2	1.834	1.8329	1	$\bar{5}\,3\,0$

5. Single-crystal X-ray diffraction

Single-crystal studies were carried out on the macromolecular beam line MX2 of the Australian Synchrotron. A 15 μm × 2 μm × 2 μm needle of Foote mine kayrobertsonite was used for data collection, using an ADSC Quantum 315r detector and monochromatic radiation with a wavelength of 0.71086 Å. The crystal was maintained at 100 K in an open-flow nitrogen cryostream. A φ scan was employed, with frame widths of 1° and a counting time per frame of 1 s. The data were integrated in $P1$ using XDS (Kabsch, 2010) and the absorption correction was carried out with SADABS (Bruker, 2001). Refined single-crystal unit-cell parameters at 100 K are: $a = 10.049(2)$, $b = 10.205(2)$, $c = 6.083(1)$ Å, $\alpha = 91.79(3)$, $\beta = 99.70(3)$, $\gamma = 98.02(3)$, $V = 607.9(2)$ Å^3 and $Z = 2$.

A structure solution for kayrobertsonite was obtained in space group $P\bar{1}$ using SHELXT (Sheldrick, 2015). The structural model was found to be closely related to that of nordgauite (Birch *et al.*, 2011), but with an extra water molecule located in channels in the

Table 3. Data collection and structure refinement details for Foote mine kayrobertsonite.

Diffractometer	ADSC Quantum 315r detector
X-ray radiation/power	$\lambda = 0.71086$ Å
Temperature	100 K
Structural formula	$MnAl_2(PO_4)_2(OH)_2 \cdot 6H_2O$
Space group	$P\bar{1}$
Unit-cell dimensions	$a = 10.049(2)$ Å $\alpha = 91.79(3)°$
	$b = 10.205(2)$ Å $\beta = 99.70(3)°$
	$c = 6.083(1)$ Å $\gamma = 98.02(3)°$
	$V = 607.9(2)$ Å^3
Z	2
Absorption coefficient	1.59 mm^{-1}
$F(000)$	418
Crystal size	$15 \times 2 \times 2$ μm
θ range	2.68 to 32.49°
Index ranges	$-14 \leq h \leq 13$, $-13 \leq k \leq 13$, $-8 \leq l \leq 8$
Reflections collected/unique	7467/3047 [$R_{int} = 0.12$]
Reflections with [$I > 3\sigma(I)$]	1639
Refinement method	Full-matrix least-squares on F^2
Parameters refined	194
Final R indices [$I > 3\sigma(I)$]	$R_{obs} = 0.071$, $wR_{obs} = 0.070$
R indices (all data)	$R_{obs} = 0.088$, $wR_{obs} = 0.190$
Goodness of fit	1.61

structure. The structure was refined using JANA2006 (Petříček & Dušek, 2006). Refinement employing anisotropic displacement parameters converged at $R_{obs} = 0.07$ for 1639 observed reflections with $I > 3\sigma(I)$. Details of the data collection and structure refinement are provided in Table 3. Fractional coordinates and anisotropic atom displacement parameters are given in Table 4. Selected bond distances are reported in Table 5.

Table 4. Atom fractional coordinates and displacement parameters (Å^2) for Foote mine kayrobertsonite.

	x	y	z	U_{eq}	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mn	0.58986(11)	0.42095(10)	0.35167(15)	0.0140(3)	0.0138(5)	0.0171(5)	0.0122(4)	0.0043(3)	0.0029(3)	0.0029(3)
P1	0.89520(17)	0.73003(15)	0.7087(2)	0.0119(4)	0.0128(8)	0.0126(8)	0.0102(5)	0.0024(5)	0.0009(5)	0.0019(5)
P2	0.66781(18)	0.44768(15)	0.9240(2)	0.0126(4)	0.0111(8)	0.0144(8)	0.0118(6)	0.0009(5)	0.0011(5)	0.0030(5)
Al1	0	½	0	0.0112(7)	0.0098(13)	0.0137(13)	0.0106(9)	0.0025(9)	0.0022(9)	0.0021(8)
Al2	0	1	½	0.0148(8)	0.0147(14)	0.0157(14)	0.0135(10)	0.0015(10)	0.0010(10)	0.0019(8)
Al3	0.8030(2)	0.71886(18)	0.1861(3)	0.0117(5)	0.0124(9)	0.0129(9)	0.0100(7)	0.0028(6)	0.0011(6)	0.0018(5)
O1	0.8024(5)	0.6914(4)	0.4846(7)	0.0141(13)	0.016(2)	0.016(2)	0.0113(16)	0.0015(16)	0.0038(16)	0.0027(14)
O2	0.8037(5)	0.7458(4)	0.8815(6)	0.0144(13)	0.015(2)	0.019(2)	0.0074(15)	0.0005(16)	−0.0028(15)	0.0029(13)
O3	0.9900(5)	0.8615(4)	0.6982(7)	0.0153(13)	0.016(2)	0.013(2)	0.0148(17)	0.0000(16)	−0.0018(17)	0.0012(14)
O4	0.9880(5)	0.6241(4)	0.7721(7)	0.0143(13)	0.014(2)	0.016(2)	0.0139(17)	0.0032(16)	0.0025(16)	0.0042(14)
O5	0.8115(5)	0.4234(4)	0.8944(7)	0.0141(13)	0.010(2)	0.018(2)	0.0141(17)	0.0021(16)	0.0015(16)	−0.0003(14)
O6	0.5931(5)	0.3268(4)	0.0206(7)	0.0157(13)	0.012(2)	0.020(2)	0.0144(17)	0.0023(16)	0.0011(16)	0.0076(15)
O7	0.5876(5)	0.4784(4)	0.6970(7)	0.0153(13)	0.014(2)	0.014(2)	0.0165(18)	0.0018(16)	−0.0017(17)	0.0015(14)
O8	0.6763(5)	0.5608(4)	0.1076(7)	0.0136(12)	0.013(2)	0.014(2)	0.0131(16)	0.0006(15)	0.0020(16)	0.0010(14)
OH1	0.9088(5)	0.8870(4)	0.2560(6)	0.0138(13)	0.017(2)	0.014(2)	0.0098(16)	−0.0012(16)	0.0022(16)	0.0011(13)
OH2	0.9501(5)	0.6197(4)	0.2010(6)	0.0133(13)	0.010(2)	0.016(2)	0.0127(17)	0.0024(16)	0.0001(16)	−0.0008(14)
OW1	0.8260(6)	0.0355(5)	0.5598(8)	0.0211(15)	0.018(3)	0.026(3)	0.019(2)	0.0059(19)	0.0019(18)	0.0040(17)
OW2	0.6366(5)	0.7986(5)	0.1774(7)	0.0162(13)	0.011(2)	0.019(2)	0.018(2)	0.0029(16)	−0.0006(16)	0.0020(15)
OW3	0.4491(6)	0.2338(5)	0.3793(8)	0.0201(14)	0.026(3)	0.017(2)	0.021(2)	0.0072(18)	0.009(2)	0.0031(16)
OW4	0.7794(6)	0.3486(5)	0.4504(7)	0.0223(15)	0.022(3)	0.030(3)	0.015(2)	0.006(2)	0.0016(18)	−0.0003(16)
OW5	0.8003(6)	0.1461(5)	0.9519(8)	0.0226(15)	0.024(3)	0.020(3)	0.023(2)	0.0048(19)	−0.0004(19)	0.0003(16)
OW6	0.6192(6)	0.0591(5)	0.2246(9)	0.0253(16)	0.021(3)	0.021(3)	0.032(2)	0.0033(19)	−0.001(2)	0.0007(18)

Table 5. Selected bond distances (Å) in Foote mine kayrobertsonite.

Mn−	O6	2.211(5)	Al1−	O4	1.907(4)
	O7	2.168(4)		O4	1.907(4)
	O7	2.163(5)		O5	1.938(4)
	O8	2.284(5)		O5	1.938(4)
	Ow3	2.241(5)		OH2	1.877(4)
	Ow4	2.138(6)		OH2	1.877(4)
	<Mn-O>	2.156		<Al-O>	1.907
P1−	O1	1.523(4)	Al2−	O3	1.889(4)
	O2	1.527(5)		O3	1.889(4)
	O3	1.543(5)		OH1	1.874(4)
	O4	1.540(5)		OH1	1.874(4)
	<P-O>	1.533		OW1	1.924(6)
				OW1	1.924(6)
P2−	O5	1.538(5)		<Al-O>	1.895
	O6	1.538(5)			
	O7	1.541(4)	Al3−	O1	1.847(4)
	O8	1.565(5)		O2	1.882(4)
	<P-O>	1.545		O8	1.904(4)
				OH1	1.883(4)
				OH2	1.896(5)
				OW2	1.953(6)
				<Al-O>	1.894

6. Discussion

A polyhedral representation of the structure of kayrobertsonite in projection along [001] is shown in Fig. 3. The structure comprises zig-zag strings of corner-connected Al octahedra along [011], in which the shared corners are alternately in *cis* and *trans* configuration. These chains link through corner-sharing with PO_4 tetrahedra along [001] to form (100) slabs that are interconnected along [100] into a 3D framework via corner-sharing with dimers of edge-sharing $[MnO_4(H_2O)_2]$ octahedra. Two independent water molecules, Ow5 and Ow6, occupy sites in [001] channels as illustrated in Fig. 3. The polyhedral framework in kayrobertsonite has the same
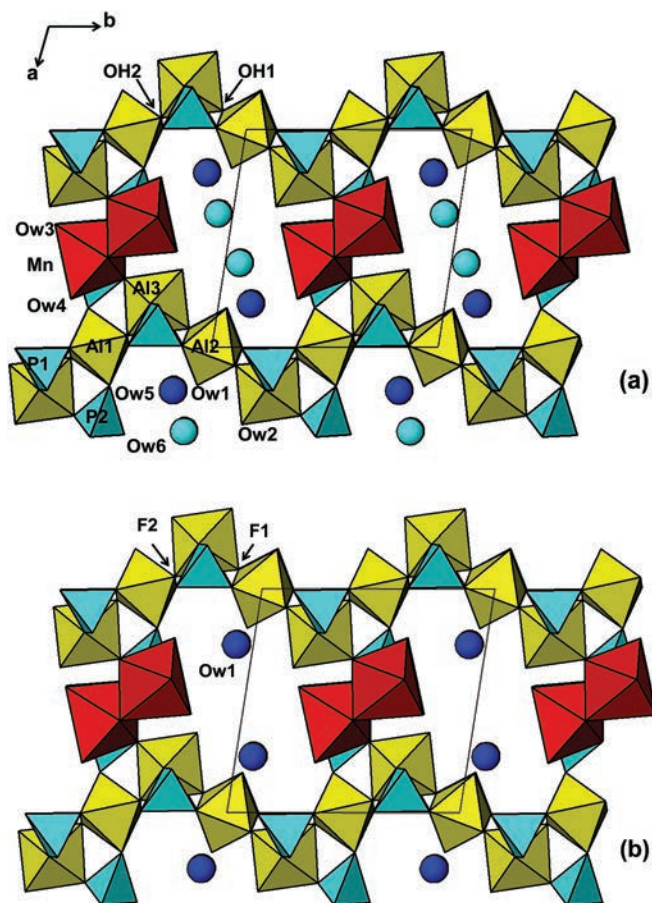

Fig. 3. Crystal structure of kayrobertsonite (a) and nordgauite (b). Water molecules are light and dark grey. Note the extra water molecule OW6 is the light grey sphere. (online version in colour)

topology as that in nordgauite (Birch *et al.*, 2011). Apart from the replacement of F by OH at the bridging anion sites OH1 and OH2 shown in Fig. 3, the main crystal-chemical change from nordgauite to kayrobertsonite is a doubling of the number of water molecules in the [001] channels. To

Table 6. Bond valence sums for Foote mine kayrobertsonite*

	Mn	Al1	Al2	Al3	P1	P2	Σ
O1				0.59	1.29		1.88
O2				0.54	1.27		1.81
O3			0.53$^{x2}\downarrow$		1.22		1.75
O4		0.50$^{x2}\downarrow$			1.23		1.73
O5		0.46$^{x2}\downarrow$				1.24	1.70
O6	0.32					1.24	1.56
O7	0.37$^{x2}\downarrow\rightarrow$					1.23	1.97
O8	0.26			0.50		1.16	1.92
OH1			0.55$^{x2}\downarrow$	0.53			1.08
OH2		0.54$^{x2}\downarrow$		0.52			1.06
Ow1			0.48$^{x2}\downarrow$				0.48
Ow2				0.44			0.44
Ow3	0.30						0.30
Ow4	0.39						0.39
Σ	2.01	3.00	3.12	3.12	5.01	4.87	

*Parameters from Brown & Altermatt (1985)

Table 7. Comparative data for nordgauite and kayrobertsonite.

	Nordgauite	Kayrobertsonite
Reference	Birch et al. (2011)	This work
Formula	$MnAl_2(PO_4)_2(F,OH)_2$ $\cdot 5H_2O$	$MnAl_2(PO_4)_2(OH)_2$ $\cdot 6H_2O$
Crystal system	triclinic	triclinic
Space Group	$P\bar{1}$	$P\bar{1}$
Synchrotron single-crystal unit cell (100 K)		
a (Å)	9.887(2)	10.049(2)
b (Å)	9.796(2)	10.205(2)
c (Å)	6.054(2)	6.0830(12)
α (°)	92.70(1)	91.79(3)
β ()	99.83(1)	99.65(4)
γ (°)	97.10(1)	98.02(3)
V (Å^3)	571.9(3)	607.9(2)
Z	2	2
D_{meas}	2.35	2.29
D_{calc}	2.48	2.41
RI	1.57 (av)	
α		1.530(1)
β		1.554(1)
γ		1.566(1)
$2V_{meas}$ (°)	nd	70.3(5)
	biaxial	biaxial
Strongest lines in X-ray powder pattern	9.806(100)	10.047(100)
	7.432(40)	7.629(44)
	4.596(12)	5.029(12)
	4.119(20)	4.695(10)
	3.225(12)	3.023(12)
	3.215(12)	2.952(10)
	2.976(12)	2.579(10)

make room for the second water molecule, Ow6, in kayrobertsonite, the water molecule Ow5 undergoes a large displacement (0.87 Å) with a closer approach to Ow1, which is coordinated to Al2. The extra water molecules in the channels of kayrobertsonite relative to nordgauite are associated with a 4.0% expansion of the unit-cell volume. The H atoms were not located in the structure refinement, but the assignments of OH⁻ and H_2O are confirmed from bond-valence calculations. These are reported in Table 6, based on the bond-valence parameters of Brown & Altermatt (1985).

The close crystal-chemical relationship between kayrobertsonite and nordgauite is shown by comparative data for the two minerals in Table 7. As mentioned above, the main differences in going from nordgauite to kayrobertsonite are OH⁻ replacing F⁻ in two sites, an increase in the channel water content and an associated unit-cell volume increase that is reflected also in a significant increase in the *d*-spacings of the powder diffraction lines.

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